

Standard Operating Procedure For Nutrient Sample Processing

LG212

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Standard Operating Procedure for Nutrient Sample Processing

1.0 SCOPE AND APPLICATION

- 1.1 This method is a description of the procedure used on the EPA monitoring vessel, the *R/V Lake Guardian* and includes the filtration and preservation of those samples to be analyzed for chloride, dissolved reactive silica, total phosphorus, nitrate plus nitrite, and total dissolved phosphorus.

2.0 SUMMARY OF METHOD

- 2.1 Water samples are collected with a Rosette Niskin bottle and transferred to a one-gallon Cubitainer. As soon as possible, usually within one to two hours, a portion of the sample is filtered and this filtrate is used to fill two 125-mL polyethylene bottles.
- 2.2 One of these two bottles and a third 125-mL polyethylene bottle of the raw sample from the cubitainer are preserved with acid equivalent to one mL/L of concentrated sulfuric acid.

3.0 SAMPLE HANDLING AND PRESERVATION

- 3.1 Samples are collected in new one-gallon cubitainers (polyethylene plastic collapsible containers).
- 3.2 These procedures are performed within two hours of collection.
- 3.3 Clean Nitrile gloves are worn while performing this procedure to prevent contamination from oils from the hands of the operators.
- 3.4 The labels for nutrient samples are color coded: the TP sample labels are yellow, the TDP/NO₃ sample labels are orange, and the Cl/Si sample labels are white.
- 3.5 The labels for nutrient samples are bar-coded so that they can be tracked and loaded into the Lachat Auto-analyzer automatically.
- 3.6 Lab and field duplicates are separate filtrations, the lab duplicate prepared from the routine field sample, the field duplicate from the duplicate sample. See WQS QAPP.

4.0 APPARATUS

- 4.1 Source of vacuum (Ship's vacuum pump, tank, and automatic cut-off).
- 4.2 Vacuum regulator set to 5 psi, with safety flask to prevent liquid from entering the regulator.
- 4.3 Three 500-mL plastic separatory funnels with two-hole rubber stoppers.
- 4.4 Three 300-mL polysulfone 47-mm filter holder with magnetic seal.

- 4.5 Three three-port two-way valves, to allow hose to top of separatory funnel to be connected to either 5 psi vacuum or open to atmosphere.
- 4.6 Filter forceps.
- 4.7 Bottle-top dispenser set to deliver 0.4 mL.
- 4.8 Supply of 47-mm diameter Sartorius 0.45- μ m membrane filters.

5.0 REAGENTS

- 5.1 Sulfuric acid solution. With continuous mixing, add 150 mL of concentrated sulfuric acid to 385 mL of reagent water.

CAUTION: Concentrated sulfuric acid will produce immediate charring of skin and natural fibers like cotton, wool and paper. Very high temperatures are generated when sulfuric acid is mixed with water. Never add water to containers of sulfuric acid. This operation should be performed in a fume hood.

6.0 EQUIPMENT CONFIGURATION

- 6.1 The vacuum regulator is connected to the ship's vacuum system with vacuum tubing. The other connections can be made with regular laboratory tubing. The regulator is connected through the safety flask to three two-way valves which are each connected to one hole of a two-hole stopper in the top of the separatory funnel, such that the separatory funnel connection can be switched between vacuum and atmosphere. The other hole in the two-hole stopper holds the filter funnel. The combination of filter funnel and separatory funnel are supported in a rack such that the separatory funnel can be conveniently drained into a 125-mL bottle.
- 6.2 The bottle-top dispenser is set to deliver 0.4 mL of the sulfuric acid solution (5.1). It must be enclosed in secondary containment and placed in a secure area to preclude being overturned by the motion of the ship or by accidental contact.

7.0 PROCEDURE

- 7.1 Clean Nitrile gloves must be worn while working with the filtering equipment.
- 7.2 Prior to each monitoring survey, the filter funnels and separatory funnels are rinsed thoroughly with tap water and a laboratory brush, followed by rinsing with reagent water. Between stations, the filter funnels are covered with aluminum foil. At the conclusion of each filtering operation, filter forceps are used to replace the dirty filter with a fresh filter.
- 7.3 For each sample, the filter cover is removed from the filter funnel, the separatory funnel stopcock is closed, the cubitainer contents are mixed by agitation and about 100 mL of sample are placed in the filter funnel. The two-way valve is turned to introduce vacuum to the separatory funnel. As soon as the sample has filtered through to the separatory funnel, the two-way valve is turned to atmosphere and the contents are used to rinse the TDP/NO₃ sample storage bottle and the Cl/Si sample storage bottle. The separatory funnel stopcock is closed, the cubitainer contents are mixed by agitation and about 250 mL of sample are added to the filter funnel. The two-way valve is turned to put vacuum on the separatory funnel. When the sample has filtered into the separatory funnel, the two-way valve is turned to atmosphere, the separatory funnel contents are

drained into the TDP/NO₃ sample storage bottle and the Cl/Si sample storage bottle. With the filter forceps, the used filter is replaced with a new filter. If there are no more samples, the cover is placed back on the filter funnel. Sometime during the filtering operation, the cubitainer is mixed by agitation and the TP sample storage bottle is rinsed with and then filled with material from the cubitainer. When all of the samples have been filtered, the bottle-top dispenser is used to add 0.4 mL of sulfuric acid to each of the TDP/NO₃ samples and TP samples in their sample storage bottles. The operator then places the nutrient sample storage bottles in a refrigerator, and records his employee ID and finish time in the Batch Log. The samples are sorted into analytical batches and placed into Ziploc bags by the GLAS contract chemist prior to being transported back to the Analytical Laboratory.

8.0 SAFETY AND WASTE HANDLING

- 8.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures; and 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.
- 8.2 All containers storing reagents, standards, controls, blanks, and wastes used in the laboratory must be properly identified through appropriate labeling and hazard definition.
- 8.3 Every chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Please refer to Appendix L in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) for more detailed descriptions of the potential risks associated with any chemicals used in this method. It is good laboratory practice to wear a lab coat, safety goggles and gloves at all times.
- 8.4 It is the responsibility of the user of this method to comply with relevant chemical disposal and waste regulations as cited in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended). All applicable safety and waste handling rules are to be followed. Good technique includes minimizing contaminated waste.
- 8.5 Over-board discharges of chemical wastes are forbidden.